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13. ABSTRACT (Maximum 200 words)

Several processing methods to improve the mechanical properties of polypropylene (PP) by blending them with liquid crystalline polymers (LCPs) have been investigated in this study. Injection molded plaques of PP blended with LCPs showed significant improvements in the modulus but did not exhibit any increase in tensile strengths and had poor surface finish. Higher tensile strength and better surface finish were obtained by compatibilization of PP with LCP. Compatibilization was carried out by a reactive extrusion process in which PP was grafted to LCP and it also further enhanced the modulus of the PP/LCP blends. Finally the compatibilized and the non-compatibilized PP/LCP blended plaques were processed by solid phase forming techniques in which the composites were deformed after preheating them below the melting point of the LCPs. This led to substantial increase in their modulus while not altering their tensile strengths.

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Processing Studies of Blends of Polypropylene

with Liquid Crystalline Polymers

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INTRODUCTION

The blending of polymers with liquid crystalline polymers (LCPs) to improve their mechanical properties has been an area of considerable interest to researchers in the last few years [1-5]. Under certain conditions, LCPs can be oriented in the blend to reinforce the matrix and thereby enhance the mechanical properties. Some of the conditions necessary to generate the fine fibrils of the reinforcing LCPs in the matrix polymer have also been established [1,6]. The LCPs usually have higher melting points and are thus processed at relatively higher temperatures than some of the commodity thermoplastics such as poly(ethylene terepthalate), PET, or polypropylene, PP. In our laboratories, we have developed a new blending technique by means of which PET and PP can be blended with a variety of LCPs [7,8]. Using this new technique, we have successfully made PP and PET composites with LCP reinforcements having higher processing temperatures than the matrix. The properties of these composites for injection molded materials match or exceed those based on glass fiber reinforced systems [9]. However, the real advantage in this form of composite may be in the processing options available to these systems but not to the glass filled systems. [7].

Preliminary studies in our laboratory on blends of PP with several LCPs have shown that the blends while exhibiting significant improvements in modulus tend to have decreased tensile strength compared to that of pure PP and poor surface finish, both of which may be attributed to their incompatibility. Compatibilization improves the dispersity and the mechanical properties of immiscible blends [10]. Block or graft copolymers possessing segments with chemical structures or solubility parameters which are similar to to those polymers being blended are effective compatibilizing agents. Insitu generation of these block or graft copolymers is possible by reaction between the blend components and these act as surfactants to lower the interfacial tension and promote improved adhesion between the phases [10]. It may thus be worthwhile to investigate whether compatibilization can lead to further improvement of the mechanical properties and even the surface finish of the PP/LCP blends.

Solid phase forming, processes by which polymers are deformed below their melting point, is another way to improve the mechanical properties of polymers. Improvements in properties of semi-crystalline polymers in more than one direction have been achieved by a solid state deformation process which involves preheating the material, passing it through heated rollers and then drawing it [11,12]. Deforming polymeric materials under compression can lead to the enhancement of mechanical properties [13] and in our laboratories, the improvement in tensile properties of pure LCPs by non-isothermal pressing was accomplished [14].

The purpose of the current study is three fold. The first goal is to examine whether tensile properties of PP can be improved by blending them with LCPs using injection molding. The next objective is to investigate the role of compatibilization, if any, in enhancing the tensile strength and modulus and improving the surface finish of PP/LCP blends processed by means of injection molding. In addition, we would like to determine the feasibility of further enhancing the mechanical properties of the LCP reinforced PP composites by solid phase forming techniques. The solid phase forming techniques, which are basically compression molding and calendering, involve deforming the composites after heating them at temperatures below the melting point of the LCP. In the first forming method, the PP composites are pressed non-isothermally in a combination of hot and cold presses. In the second method, the PP/LCP composites are preheated and pressed between calendering rolls.

EXPERIMENTAL

Materials The two LCPs used for this study were purchased from Hoechst Celanese. Both of them are marketed commercially under the brand name Vectra. The first LCP, Vectra A900, is a co-polyester of p-hydroxy benzoic acid and 6-hydroxy-2-naphthoic acid. The second LCP is a polyester (coamide) and is called Vectra B950. Its possible structure can be found in reference [5]. Vectra A900 and Vectra B950 will henceforth be referred to as Vectra A and Vectra B, respectively. The matrix PP, Profax 6823, was supplied by Himont.

Before processing, pellets of the LCPs were dried for at least 24 hours in a convection oven at 110 °C. The dried pellets were then tumbled together with PP pellets in a predetermined weight ratio for making blended injection molded plaques. Injection molded plaques containing 20 and 30 wt % LCP were made for PP/Vectra A and PP/Vectra B blends. The compatibilized PP/Vectra B blended plaques contained 20 and 30 wt % Vectra B. Compatibilization of PP/LCP was carried out by a reactive extrusion process in which the PP was grafted to the LCP [15].

Injection Molding Rectangular plaques of approximate dimensions 75 mm by 80 mm and 1.75 mm thickness were injection molded using an Arburg model 222-55-20. The barrel temperatures of the extruder in the injection molder was 230 ° C in the first zone and 295 °C in the second and third zone. The nozzle temperature was 250 °C and the mold was kept at room temperature.

Solid Phase Processing The plaques were processed using two methods. In the first method, the samples were coated with Dow Corning oil (710 Fluid) and covered with Teflon sheets and then placed between steel plates. The plaques were preheated to a temperature of 155 or 165 °C for 15 minutes in a hot press without pressing. The samples together with the steel plates were then removed from the hot press and pressed in a cold press (water cooled to 10 ° C) with a 111000 N force (apprx. 4.8 MPa pressure based on the plaque surface area). Steel shims were placed between the steel plates to control the thickness reduction of the plaques.

In the second method, 25.4 mm thick strips were cut from the PP/Vectra plaques. The strips were preheated in an infrared oven and then passed through heated calender rolls which were also at the preheated temperature. Preheating (or calendering temp.) was done at 145 °C, 155 °C and 165 °C. The roller speeds were held constant at 11 mm/s. After calendering, the samples were quenched under load at room temperature.

Rheological Studies The rheological studies of PP and various PP/LCP blends were conducted on a Rheometrics Mechanical Spectrometer (RMS800). Cooling experiments were done in the dynamic mode using the parallel plate device with a gap of approximately 1.4 to 1.5 mm. In all the cooling experiments with the blends, the samples were first preheated to 290 °C and maintained there for 4 minutes before the experiments were started. The complex viscosity, the storage and loss moduli were monitored as a function of temperature. An angular frequency of 10 rad/s and a strain of 5 % were used in all the dynamic experiments.

Mechanical properties The tensile modulus and strength of the unpressed, pressed and calendered materials were measured using an Instron mechanical tester (model 4204). The test samples were strips of approximate dimensions 80 mm long and 12.5 mm wide. The cross head speed was 2 mm/min. The strain was measured using an extensiometer (MTS 632.13B.30). The arithmetic average and the standard deviation of the tensile properties were calculated using a minimum of five samples.

The dynamic torsional properties of the plaques were obtained using the RMS 800. In particular, the torsional modulus (G') was obtained as a function of temperature using an angular frequency of 10 rad/s and a strain of 0.07%. The test sample size was a rectangular piece approximately 50 mm long, 12.5 mm wide, and 1.1 to 1.6 mm thick.

Morphological Studies The morphology was determined by scanning electron microscopy (SEM), using a Cambridge Stereoscan S200 with an accelerating voltage of 25 kV. The samples were fractured after immersing them in liquid nitrogen for five minutes. The fractured samples were then placed on aluminum stubs and sputter-coated with gold using an SPI sputter coated for enhanced conductivity.

RESULTS AND DISCUSSION

Rheological studies were conducted to establish the processing temperature conditions for injection molding the plaques. Time sweeps of PP and PP/LCP blends at 295 °C in which complex viscosity and the moduli values were monitored showed that these values were stable for at least 4 minutes. This is longer than the estimated residence time at 295 °C in the injection molder. The cooling experiments of the various blends and PP shown in Fig. 1 give us some of the behavior during injection molding. The solidification of pure PP is relatively slow as observed by the nearly constant viscosity with drop in temperature until 150 °C. For the blends there appears to be a two stage solidification (crystallization) process indicated by an initial rise in complex viscosity (about 280 °C) which can be attributed to the LCP phase and then a slower solidification starting about 240 °C due to the PP phase. This indicates that even without significant preheating of the blends there is a processing window (dominated by the crystallization of the LCP phase) within which the LCP phase can still be deformed. The advantages of preheating the LCPs to higher temperatures have been discussed elsewhere [1,7,8]. The injection molding temperatures were selected such that the blends would be maintained within the processing window described above.

The tensile properties of injection molded plaques of PP/LCP blends compared to those of pure PP are shown in Table 1. For 20 wt % LCP content, both the PP/Vectra A and PP/Vectra B plaques show a two-fold increase in the tensile modulus over that of pure PP. The moduli are further enhanced when the LCP content is increased to 30 wt % in the blends. On the other hand, there is hardly any difference between the values of tensile strengths of the PP/Vectra A and PP plaques. However, the tensile strength drops substantially for the PP/Vectra B composite containing 30% by weight of LCP. The surface of the blended PP/Vectra plaques have a dull appearance and poor finish. The lack of improvements and at times decrease in the tensile strengths values coupled with the poor surface finish may be attributed to the incompatibility of the PP with the two LCPs.

Fractured surfaces of the blends were observed under the SEM to ascertain the morphology and WAXD patterns were obtained to investigate the molecular orientation in these blends. Fig. 2 shows the fractured surfaces of two of these LCP blends, containing 30 wt % Vectra A and Vectra B, scanned perpendicular to the machine direction. Both the micrographs show long LCP fibrils which appear oriented in the machine direction. WAXD patterns of the PP/LCP blends, containing 30 wt % Vectra A and Vectra B, are shown in Fig. 3. The sharp arcs were determined to correspond to the LCP (Vectra) phase which indicate their high degree of molecular orientation. The presence of these highly oriented fibrillar structures is responsible for enhancement in the properties of the blends. Similar morphology and orientation were observed for the blends containing 20 wt % Vectra A and Vectra B.

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In spite of the presence of the favorable morphology and a high degree of molecular orientation arising from the LCP phase, the tensile strengths did not increase as anticipated. This fact and the observation regarding the poor surface finish of the blends are likely to be attributed to the incompatibility of the PP with the two LCPs. Compatibilitization of the PP/Vectra blends is one possible method of overcoming the problems arising out of poor compatibility. Compatibilization also might further improve the tensile modulus of the blends which has the potential to be further enhanced when one considers the very high moduli of the reinforcing LCPs.

The effects of the compatibilization on the tensile properties of PP/Vectra B blends are presented in Table 1. There are significant improvements in the tensile moduli of the compatibilized PP/Vectra B blends over those blends which were not compatibilized. An additional 30 to 35% increase in the modulus of the blends has been achieved by compatibilization, as compared to those of the non-compatibilized PP/Vectra B blends. Overall about 2.5 to 3.2 fold increase in the tensile properties for the 20 and 30 wt % LCP blends, respectively, over that of pure PP has been obtained. The tensile strengths of the PP/Vectra B blends dropped below those of pure PP, but on compatibilization the tensile strength recovered and was further enhanced beyond that of the PP matrix. The enhanced modulus and strength suggest possibly better adhesion between PP and Vectra B. It is believed that the compatibilization, occurring during the reactive extrusion process brings, about grafting of PP with Vectra B and this improves their mutual compatibility and the adhesion between PP and Vectra B.

It should be noted that there is a dramatic difference in the surface of the blends with and without compatibilizers. Unlike the blends without compatibilizers, the compatibilized blends have a very smooth and polished surface. Also, their appearance is much more homogenous.

The morphology of the blends, with and without compatibilization, containing 20 wt % Vectra B are shown in Figs. 4 and 5. The two morphologies look similar with the presence of the reinforcing fibrils. Although not quite apparent, the fibrils seem to be more evenly distributed and not bunched together in the compatibilized blend, as compared to the non-compatibilized blend. This indicates that the fibrils were possibly better dispersed in the former case which is usually expected of compatibilized blends. Further studies regarding the structural difference between the compatibilized and the non-compatibilized blends are being carried out.

Having reinforced the PP with LCP, and having achieved additional improvements in tensile properties and surface finish by compatibilization, we now investigate the effect of two forming techniques for further improvements in the properties of the PP/Vectra B blends. Non-isothermal compression molding was performed on one compatibilized (PP/Vectra B) blend and one blend without compatibilizer (PP/Vectra A). The PP/Vectra A blends were calendered to improve their properties.

The effect of non-isothermal compression molding at different preheating temperatures for the two blends are summarized in Tables 2 and 3. From Table 2 it is observed that for the compatibilized PP/Vectra B composites containing 20 wt % LCP, the modulus increases when pressed at room temperature after preheating at both 155 and 165 °C. For the 30 wt % PP/Vectra B blend, the modulus also increases when samples are pressed after preheating them to 155 °C, and the modulus is further increased when the preheating temperature is raised to 165 °C. The moduli of the compatibilized blends are increased by 20 to 25% on non-isothermal compression molding, and overall there is almost a three to four fold increase in modulus over that of pure PP. Similar trends are observed for the non-compatibilized PP/Vectra A blends that were pressed non-isothermally. As seen in Table 3, there is a 20 to 30% increase in the moduli on pressing the PP/Vectra A plaques and substantial increase (up to 3.5 fold) from that of pure

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PP. The tensile strengths of the blends were not altered by pressing. The morphologies of the unpressed and the pressed plaques (pressed after preheating at 165 °C) of the compatibilized PP/Vectra B, containing 30 % LCP, are compared in Fig. 6 and show no noticeable differences. The same is true for the morphologies of other unpressed and pressed PP/Vectra A and PP/Vectra B samples and thus these are not compared here. It is possible that further orientation of the LCP phase and the amorphous phase of the PP takes place while deforming and these orientations are locked in during the rapid cooling process in the cold press. The newly formed orientations can possibly lead to enhancement of mechanical properties and are the subject of current investigation.

The torsion modulus versus temperature curves for the PP/Vectra blends in Fig. 7 can be used to determine the temperatures to which the PP/Vectra blends need to be preheated before effectively compressing them in order to improve their properties. The improvement in property by preheating the blended plaques to 155 or 165 °C and then compressing in a cold press has already been discussed. On the other hand it was observed that when the plaques were preheated to 145 °C and pressed, there was hardly any reduction in thickness and consequently no property modifications occurred. The critical temperature, for preheating before deformation can be induced in the blends, seems to be the onset of softening of the dynamic torsion modulus in Fig. 7. Thus it is necessary to preheat the plaques above this critical temperature before they can be deformed to improve their properties.

The non-isothermal compression molding studies were done to establish the feasibility of other forming techniques such as calendering and thermoforming. The results of preheating and calendering the PP/Vectra A plaques at different preheating and calendering temperatures are shown in Table 4. The percentage reduction in thickness are of the same order of magnitude as those from the non-isothermal compression molding. Still, there was no change in the mechanical properties of the calendered composites. This could be owing to several factors such as low plaque temperature during calendering and lower pressure between the calender rolls. It might be possible that the hot rolls were able to produce oriented structures which were not retained and relaxed in the absence of a quick quenching process after calendering.

CONCLUSIONS

Several processing methods that can be used to substantially improve the mechanical properties of PP by blending them with two different LCPs have been reported. The blending of LCPs with PP, compatibilizing them and further processing them by solid phase forming techniques, in steps, are able to enhance the modulus of the PP/LCP composites to values approximately 3.5 to 4.0 times over that of pure PP. Compatibilization also improves the surface finish and the tensile strengths of the PP/Vectra blends, which were retained during solid phase forming.

It is clear from the results that the PP reinforced with LCP has the advantage of not only significantly enhanced properties but also a range of processing options. In addition to the ones described here, blow molding and thermoforming are also being carried out and will be reported in later publications There is potential for further property enhancement by optimizing the various processes. Also the use of the new blending technique developed in our laboratories [7,8] will allow for preheating the LCPs to higher temperatures and can generate better distribution of the LCP phase and continuous nature of the reinforcements will lead to even higher properties.

ACKNOWLEDGEMENT

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Table 1 Tensile Properties of PP/Vectra A & PP/Vectra B Composites

Materials	Tensile Modulus GPa	Tensile Strength MPa
PP	1.354 (0.075)	26.89 (1.10)
PP/Vectra A 80/20	2.555 (0.216)	27.70 (1.06)
PP/Vectra A 70/30	2.868 (0.280)	28.26 (1.15)
PP/Vectra B 80/20	2.553 (0.169)	24.04 (1.14)
PP/Vectra B 70/30	3.003 (0.289)	19.71 (0.59)
PP/Vectra B 80/20 (compatibilized)	3.209 (0.262)	34.01 (1.06)
PP/Vectra B 70/30 (comatibilized)	4.101 (0.401)	37.55 (3.47)

Standard Deviations are given in parenthesis.

Table 2   Tensile Properties of Non-Isothermally pressed PP/Vectra B Composities (Compatibilized)

Materials	Process. Cond. (°C) Heat. Temp./ Press Temp.	Percent Thickns. Reductn.	Tensile Modulus GPa	Tensile Strength MPa
PP	N.P.	-	1.354 (0.075)	26.89 (1.10)
PP/Vectra B 80/20 (C)	NP	-	3.209 (0.262)	34.01 (1.06)
PP/Vectra B 80/20 (C)	155/20	4.7	3.945 (0.282)	35.53 (1.95)
PP/Vectra B 80/20 (C)	165/20	21.0	3.885 (0.406)	34.23 (0.95)
PP/Vectra B 70/30 (C)	NP	-	4.101 (0.401)	37.55 (3.47)
PP/Vectra B 70/30 (C)	155/20	6.1	4.780 (0.252)	38.37 (1.02)
PP/Vectra B 70/30 (C)	165/20	17	5.297 (0.300)	39.23 (2.23)

Standard Deviations are given in parenthesis.
NP: non-pressed
(C) : Compatibilized

Table 3   Tensile Properties of Non-isothermally pressed PP/Vectra A Composities

Materials	Process. Cond. (°C) Heat. Temp./ Press Temp.	Percent Thickns. Reductn.	Tensile Modulus GPa	Tensile Strength MPa
PP	N.P.	-	1.354 (0.075)	26.89 (1.10)
PP/Vectra A 80/20	NP	-	2.555 (0.216)	27.70 (1.06)
PP/Vectra A 80/20	155/20	5.2	3.244 (0.155)	25.32 (0.42)
PP/Vectra A 80/20	165/20	13.2	3.762 (0.279)	28.08 (0.48)
PP/Vectra A 70/30	NP	-	2.868 (0.280)	28.26 (1.15)
PP/Vectra A 70/30	155/20	5.6	3.369 (0.181)	24.10 (0.88)
PP/Vectra A 70/30	165/20	12.0	4.660 (.125)	25.53 (0.63)

Standard Deviations are given in parenthesis.
NP: non-pressed

Table 4 Tensile Properties of Calendered PP/Vectra A Composities

Materials	Calender & Preheat. Temp. °C	Percent Thickns. Reductn.	Tensile Modulus GPa	Tensile Strength MPa
PP	N.C.	-	1.354 (0.075)	26.89 (1.10)
PP/Vectra A 80/20	NC	-	2.555 (0.216)	27.70 (1.06)
PP/Vectra A 80/20	145	5.5	2.479 (0.346)	23.29 (0.78)
PP/Vectra A 80/20	155	9.1	2.425 (0.181)	22.58 (1.00)
PP/Vectra A 80/20	165	11.3	2.556 (0.367)	22.87 (0.69)
PP/Vectra A 70/30	NC	-	2.868 (0.280)	28.26 (1.15)
PP/Vectra A 70/30	145	6.3	3.042 (0.227)	20.94 (0.53)
PP/Vectra A 70/30	155	8.5	2.821 (0.170)	22.70 (1.02)
PP/Vectra A 70/30	165	11.7	2.999 (.263)	21.05 (1.27)

Standard Deviations are given in parenthesis.
NC: non-Calendered

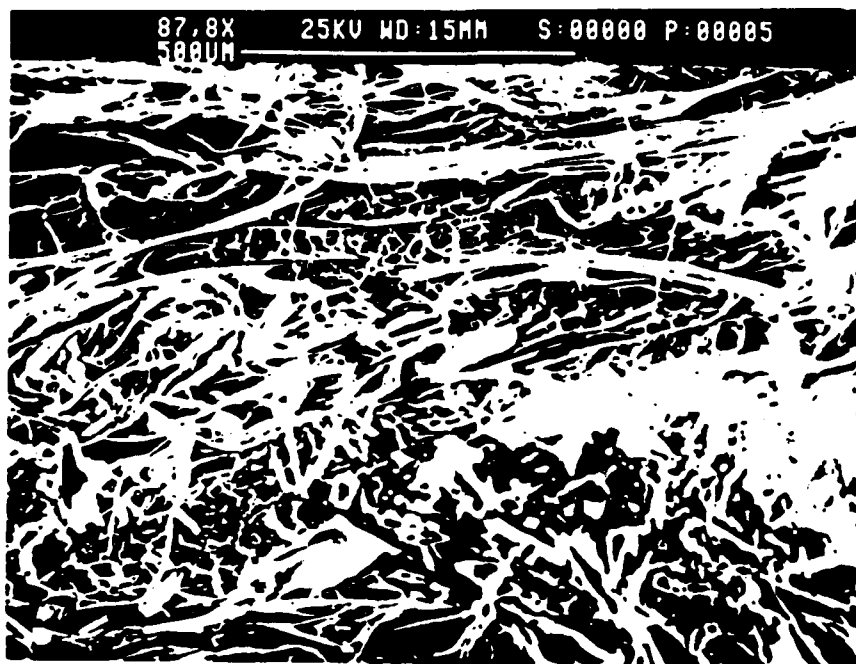Fig. 1 Complex Viscosity versus temperature for PP, PP/Vectra B and compatibilized PP/Vectra B blends.

a) PP
b) PP/Vectra B 8/2
c) PP/Vectra B 8/2 (Comp.)
d) PP/Vectra B 7/3
e) PP/Vectra B 7/3 (Comp.)

Temperature °C

Complex Viscosity [Pa.s] (Δ)
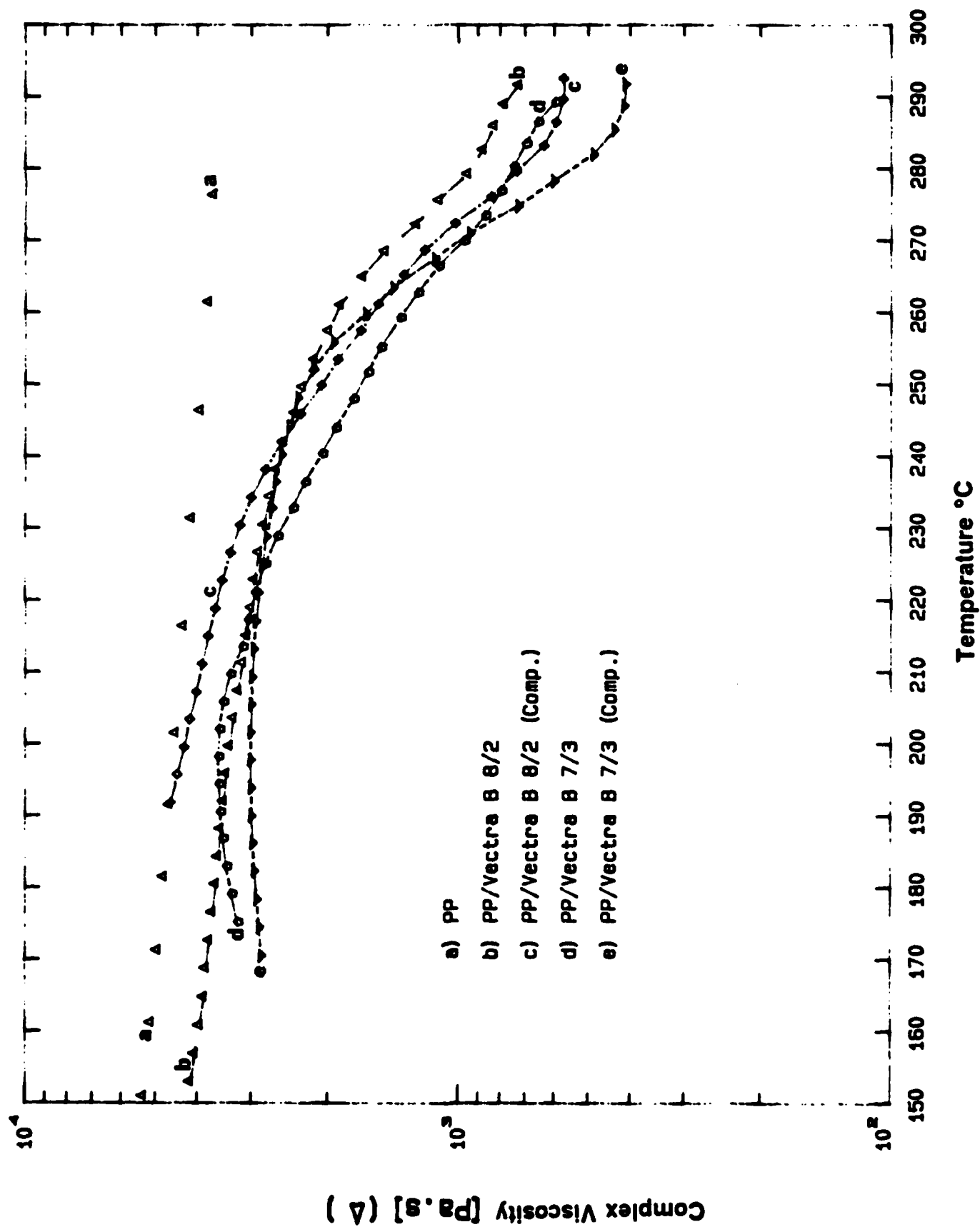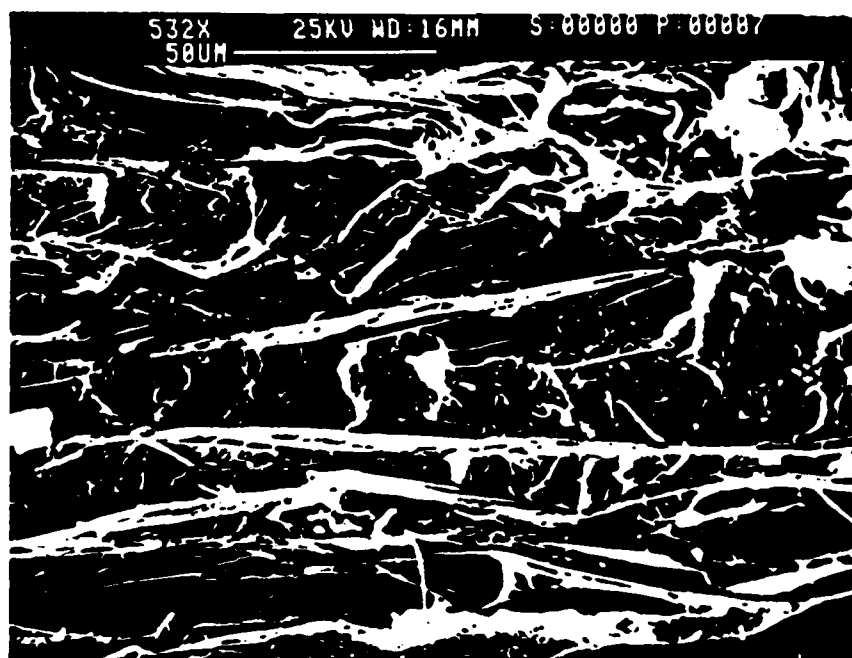
Fig. 2 SEM micrographs of PP/Vectra A 70/30 (top) and PP/Vectra B 70/30 (bottom) plaque surfaces fractured along the machine direction.
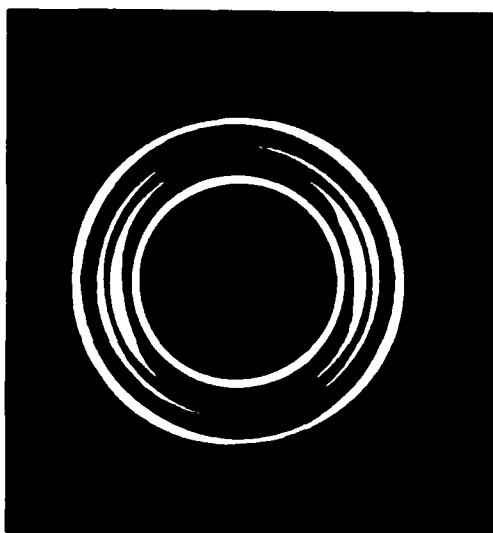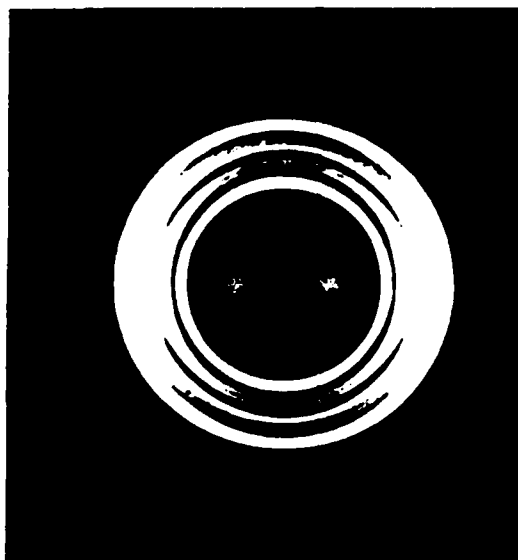
Fig. 3 WAXD patterns of injection molded PP/Vectra A 70/30 blends (top) and PP/Vectra B 70/30 blend (bottom) The machine direction is vertical.
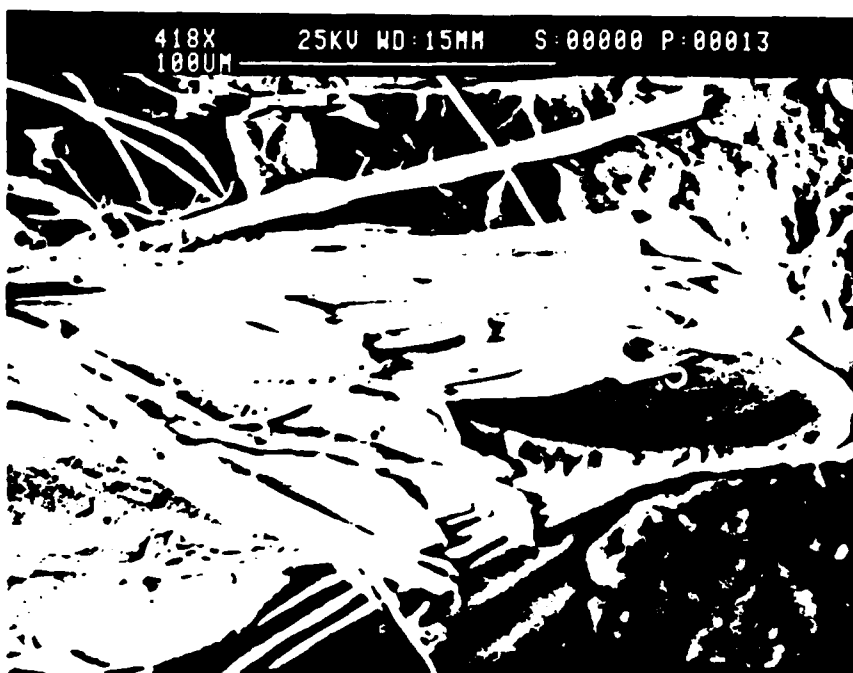
Fig.4 SEM micrographs of PP/Vectra B 80/20 plaque surfaces fractured along the machine direction.
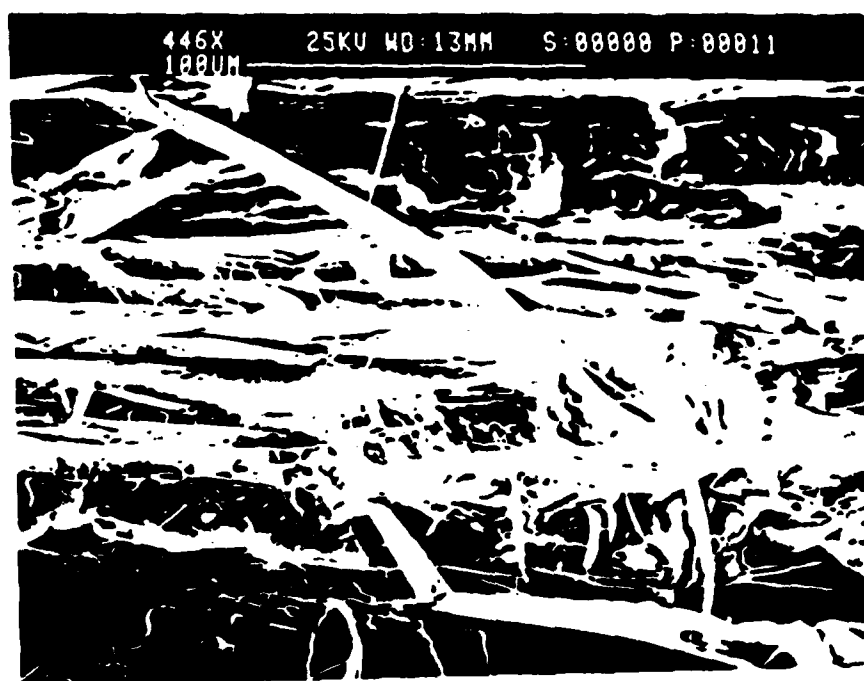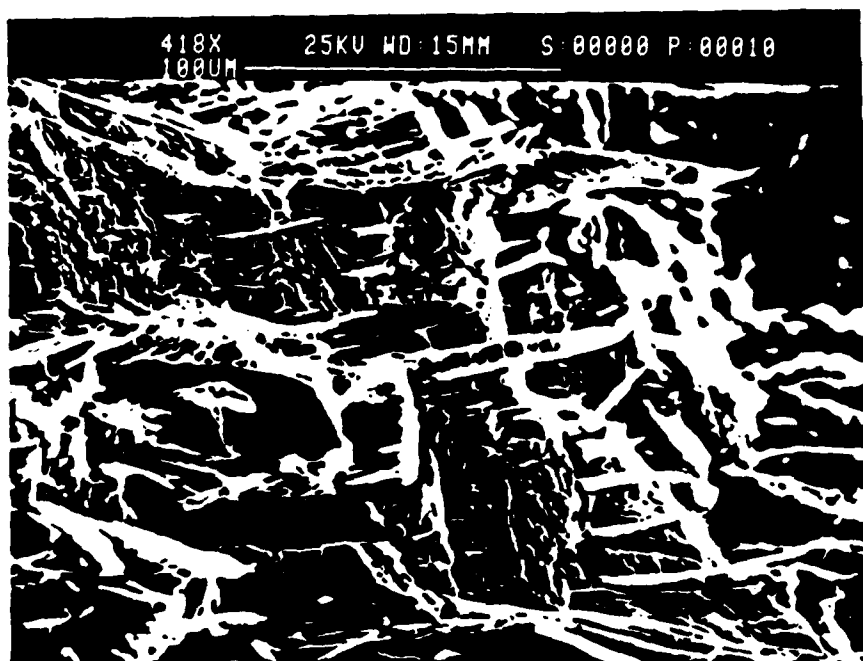
Fig.5 SEM micrographs of PP/Vectra B 80/20 (compatibilized) plaque surfaces fractured along the machine direction.
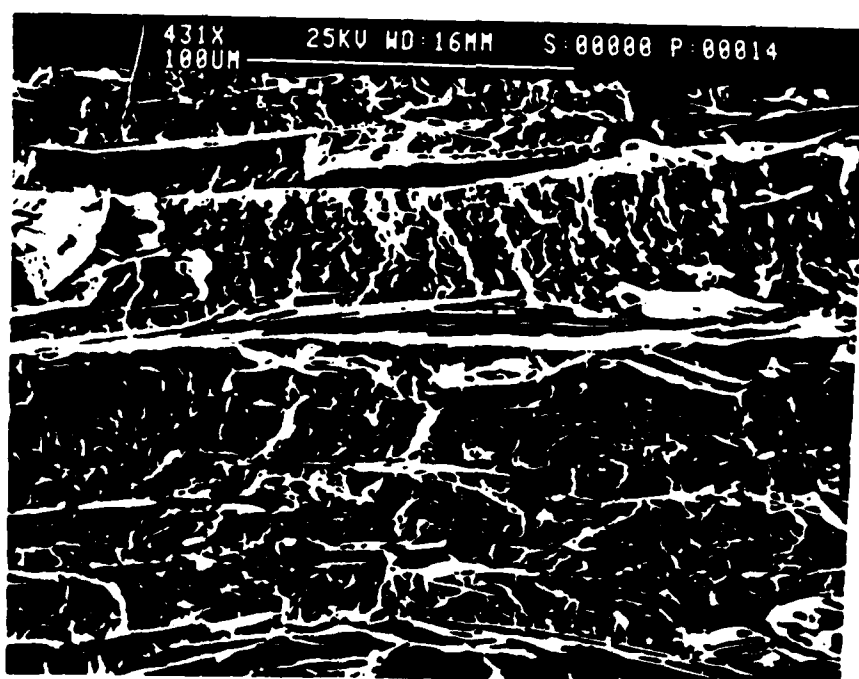
Fig.6 SEM micrographs of unpressed (top) and pressed (bottom) PP/Vectra B 70/30 (compatibilized) plaque surfaces fractured along the machine direction. The pressed plaques were heated at 165°C before pressed at 20 °C.

Fig. 7 Dynamic Torsional Modululus vs Temperature of PP/Vectra A and compatibilized PP/Vectra B Blends.

a) PP/Vectra B 7/3 (Comp.)
b) PP/Vectra B 8/2 (Comp.)
c) PP/Vectra A 7/3
d) PP/Vectra A 8/2
e) PP